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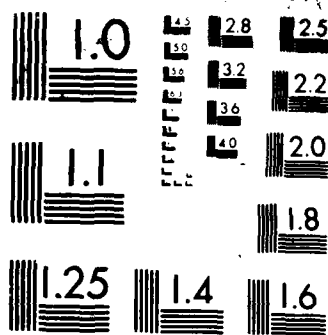
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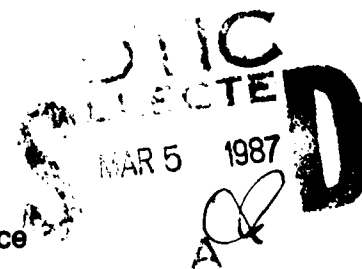
**MICROFABRICATED STRUCTURES FOR THE  
MEASUREMENT OF ADHESION AND MECHANICAL  
PROPERTIES OF POLYMER FILMS**

by

**Mark G. Allen and Stephen D. Senturia**

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# MICROFABRICATED STRUCTURES FOR THE MEASUREMENT OF ADHESION AND MECHANICAL PROPERTIES OF POLYMER FILMS

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## Introduction

Determination of the mechanical properties and adhesive strength of thin films in microelectronic devices is important both during fabrication and in evaluation of long term device reliability. Many tests of these properties are available [1-5], but few combine the advantages of an *in-situ* measurement technique and compatibility with standard integrated circuit processes. Several types of microfabricated structures for the measurement of the mechanical properties of polymer films have been fabricated in our laboratory [6,7]; the structures discussed in this work are suspended (free-standing) square membranes of a polymer film supported on an oxidized silicon wafer. In this paper, we report the use of these structures for the *in-situ* measurement of adhesion of polymer films.

## Sample Fabrication

Suspended membranes are made by first fabricating a square diaphragm 5 microns in thickness in an oxidized silicon wafer using photolithography and anisotropic etching techniques [6]. The wafer is then spin-coated with the polymer of interest and the diaphragm is removed with a backside plasma etch to create a free-standing polymer membrane. Square membranes of a BTDA-ODAMPDA polyimide (cast without adhesion promoter) from 1 to 25 millimeters (mm) on a side and ranging from 8 to 15 microns in thickness have been fabricated using this technique.

Alternate structures which are based on the suspended membranes are 'island' structures. To fabricate this structure, the square silicon diaphragm is defined so as to leave a small island of thick silicon at the center. The polymer is then spin-cast and cured as for the suspended membranes. Upon removal of the silicon diaphragm, the suspended membrane is left with a small silicon island adhered to its center. This structure is then used as the basis for further adhesion tests.

## Mechanical Property Measurements

The residual stress and Young's modulus of the film can be determined by measurement of the load-deflection behavior of the membrane (see Fig. 1) [3,7,8]. The wafer is epoxied to a substrate which seals the cavity under the membrane and placed in a chuck which permits the application of differential pressure by use of either a pressure source or a microliter syringe. The differential pressure is measured using a silicon pressure transducer mounted in the chuck. The entire assembly is placed on a microscope stage with a calibrated z-axis and the deflection  $d$  of the film at the center of the membrane is measured.

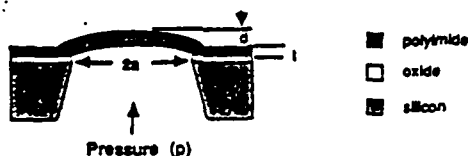


Figure 1. Membrane parameters

A theoretical analysis of the load-deflection behavior has been performed using membrane mechanics (the energy minimization approach of Timoshenko [9]), modified to account for the presence of residual stress [10]. This leads to the following relation:

$$\left(\frac{Et}{a^4}\right) d^3 + \left(\frac{1.661 \sigma_0}{a^2}\right) d = 0.547 p \quad (1)$$

where  $p$  is the applied pressure,  $E$  is Young's modulus,  $\sigma_0$  is the residual stress in the film,  $2a$  is the length of the square side,  $t$  is the film thickness, and  $d$  is the deflection at the center of the membrane. Using this approach, we have previously found values of  $E=3$  GPa and  $\sigma_0=30$  MPa for this polyimide [7]. Knowledge of both the above equation and the mechanical property data are necessary for the study of polymer adhesion using these structures.

### Adhesion Measurements - Suspended Membranes

The suspended membranes can be used for a measurement of the work of adhesion  $\gamma_a$  of the polymer film to the silicon dioxide substrate. By increasing the differential pressure on the test site, the film will peel off the substrate, forming a blister. Computer simulations of the blister volume or radius as a function of critical pressure at constant  $\gamma_a$  indicate that the blister pressure-volume characteristic is unstable; once peel has been initiated at a fixed pressure, the blister will grow without bound. This phenomenon has been observed experimentally in previous applications of the blister test [11]. Our experiment uses a controlled-volume loading to initiate and limit peel. This is accomplished by injecting pressurizing fluid (air) into the space under the blister using a calibrated microtiter syringe. The PV work necessary to peel (and stretch) the blister from its initial to final radius is measured. The injected fluid is then withdrawn, and the portion of the PV work that went into stretching the blister is measured. This procedure is illustrated graphically in Figure 2; the shaded area is the average  $\gamma_a$  times the total area peeled.

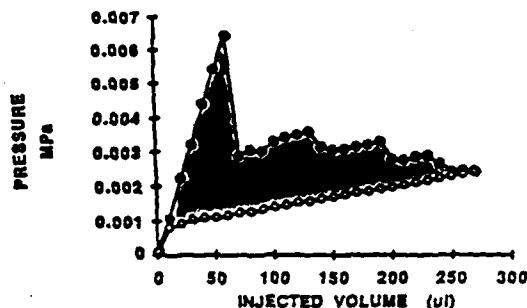


Figure 2. Adhesion PV data. Solid circles - during peel; open circles - after peel.

We have also carried out a theoretical derivation of the critical pressure necessary to initiate peeling of thin films of various geometries under lateral loading (pressure) and residual tensile stress [10]. This will be useful as a counterpart to the PV analysis described above, and is used to calculate a lower bound for  $\gamma_a$  should it be impossible to nucleate blisters without film failure. The approach utilizes a fracture energy balance and requires knowledge of the pre-peel load-deflection behavior of the suspended membrane. From an energy minimization analogous to the derivation of equation (1), the load-deflection behavior of films of several geometries with residual tensile stress can all be described by the equation:

$$p = k_1 d^3 + (k_2 + k_3) d \quad (2)$$

where  $k_1$ ,  $k_2$ , and  $k_3$  are functions of the geometry of the test site and the type of film (plate or membrane). For thin films on infinitely rigid substrates, the peel criterion is:

$$\gamma_a = \left[ \frac{5}{2} \frac{\Delta^4 c_1}{a^{11}} + \frac{3\Delta^2 c_2}{a^7} + \frac{2\Delta^2 c_3}{a^5} \right] \left[ \frac{da}{dA} \right] \quad (3)$$

where  $\Delta$  is the generalized load-point displacement of the blister (in this case, blister volume),  $da/dA$  is the incremental dependence of blister size on blister area, and  $c_1$ ,  $c_2$ , and  $c_3$  are mechanical-property-dependent constants. Table I gives the values of the various parameters in equations (2) and (3) for three blister geometries: the clamped circular plate, the circular membrane, and the square membrane. By substitution of the appropriate constants into equation (3), and simultaneous solution of (3) with the corresponding load-deflection relation (2), a value for  $\gamma_a$  can be determined as a function of the critical debond pressure  $p_c$ . Thus, experimental measurement of the critical debond pressure can be related to  $\gamma_a$  once the mechanical properties of the film have been accurately determined.

Under certain conditions, equation (3) reduces to special cases which have been previously reported in the literature. For example, in the case of a clamped circular plate with zero residual stress undergoing small deflections, ( $c_1 = c_3 = 0$ ) the peel criterion from equation (3) is:

$$\gamma_a = 0.5 p_c d_c \quad (4)$$

Table 1. Geometric constants for adhesion model

	SQUARE MEMBRANE	CLAMPED CIRCULAR PLATE	CIRCULAR MEMBRANE
$k_1$	$1.83 E t / a^4$	$2.77 E t / a^4$	$3.56 E t / a^4$
$k_2$	0	$5.68 E t^3 / a^4$	0
$k_3$	$3.041 c_0 / a^2$	$41 c_0 / a^2$	$41 c_0 / a^2$
$c_1$	$0.429 E t$	$2.42 E t$	$0.917 E t$
$c_2$	0	$5.44 E t^3$	0
$c_3$	$1.881 c_0$	$3.621 c_0$	$2.851 c_0$
$\Delta$	$16 a^2 d / \pi^2$	$d^2 d \pi / 3$	$d^2 d \pi / 2$
$da/dA$	$1/2a$	$1/2\pi a$	$1/2\pi a$

where  $d_c$  is the critical center deflection at which debond initiates; this relation has been obtained by Williams [11]. Alternatively, for the case of a circular membrane undergoing large deflections with zero residual stress, ( $c_2 = c_3 = 0$ ) the peel criterion from equation (3) is:

$$\gamma_a = 0.625 p_c d_c \quad (5)$$

Gent [12] has also analyzed this case assuming a slightly different load-deflection profile and has obtained a value of 0.65 for the premultiplying factor in equation (5).

For square test sites under residual stress such as the suspended membranes, the relation between  $\gamma_a$  and the critical center deflection  $d_c$  at which debond initiates is given by:

$$\gamma_a = 3.70 E t (d_c/a)^4 + 4.94 c_0 t (d_c/a)^2 \quad (6)$$

The relation between  $\gamma_a$  and  $p_c$  can be obtained by simultaneous solution of equations (1) and (6), allowing determination of  $\gamma_a$  from a measurement of either  $p_c$  or  $d_c$  during peel.

The upper limit of  $\gamma_a$  which can be measured using this technique is limited by the tensile strength of the film (this effect is common in many standard adhesion tests; for example, the 90° peel test is also tensile-strength limited). It was determined that membranes fabricated by the standard process and cure schedule could not be peeled; films always ruptured before blisters were formed. For these samples, a lower bound for  $\gamma_a$  was calculated by using the pressure at which the film ruptured as the  $p_c$  value for the square membrane.

In order to observe blister nucleation, it was necessary to degrade the adhesion of the  $PVSiO_2$  interface. This was done by immersing the test sites in 90°C  $H_2O$  for varying lengths of time. Table II gives a summary of the adhesion data obtained from the suspended membranes. Sample 1 was not subjected to any degradative processing and burst before blister nucleation; a lower bound for  $\gamma_a$  was calculated from equation (6) to be 360 J/m<sup>2</sup>. As expected, adhesive strength generally decreased with increased immersion time (samples 2-6) although this effect was not investigated quantitatively. Upon drying, an increase in adhesive strength was observed (samples 2,3). Agreement between the PV method and equation (6) was observed to be within 50% except for sample 4, which showed significant plastic deformation, invalidating the PV analysis. Equation (6) uses only one data point to calculate  $\gamma_a$ , while the PV method is averaged over the entire wafer. This may account for differences in the two approaches. Furthermore, equation (6) implicitly assumes an incrementally symmetric peel, which is not strictly correct for the square membrane. However, as peel of the square membrane continues, a circular blister is formed, allowing application of the appropriate form of equation (3). These details are presently under study.

#### Adhesion Measurements - Island Structures

The suspended membrane blister test, like other peel tests, is limited by the tensile strength of the film. One way to overcome this problem is to use thicker films [13]. In the blister test, we have additional flexibility. Different geometries for the microfabricated site are possible which can facilitate peel of thinner films even in systems with very good adhesion.

Equation (3) suggests that if a geometry can be found in which  $da/dA$  can be increased, larger values of  $\gamma_a$  may be measured at the same load. For simple blisters, this derivative is inversely proportional to the membrane size (Table I). Decreasing the membrane size fails since the deflection

Table II. Adhesion data

s	Sample	Environmental Conditions	Analysis method	$\gamma_a$ (J/m <sup>2</sup> )
1	8x8 blister site	none	Equation (6)	>360
2	3x3 blister site	18 hr. H <sub>2</sub> O 90°C	PV	1.5x10 <sup>-4</sup>
		18 hr. H <sub>2</sub> O 90°C + 1 hr. dry 80°C	Equation (6)	4.4x10 <sup>-3</sup>
			PV	3.8x10 <sup>-3</sup>
3	8x8 blister site	20 hr. H <sub>2</sub> O 90°C + 6 hr. dry 25°C	PV	2.0
			Equation (6)	3.1
4	10x10 blister site	8 hr. H <sub>2</sub> O 90°C	PV	1200 (*)
			Equation (6)	320
5	10x10 blister site	14.5 hr. H <sub>2</sub> O 90°C	Equation (6)	64
6	10x10 blister site	14.5 hr. H <sub>2</sub> O 90°C	Equation (6)	43

(\*) Extensive plastic deformation observed

$\Delta$  will also decrease. This problem is overcome in the island structure shown in Figure 3, where the polymer film will be peeled only off the center island. The deflection  $\Delta$  is a function of the difference  $a_2 - a_1$ , where  $2a_2$  is the characteristic size (edge length or diameter) of the entire suspended membrane, while  $2a_1$  is the characteristic size of the island. However, the derivative  $d\Delta/dA$  is proportional only to  $1/a_1$ . Thus, a large geometric advantage can be obtained by decreasing  $a_1$  while keeping  $a_2 - a_1$  large.

Although the critical pressure analysis for the island structures is considerably more complicated than for the simple blisters, an approximate relation between  $p_c$  and  $\gamma_a$  based on a circular geometry can be developed. For a membrane whose load-deflection behavior is dominated by residual tensile stress and which is suspended over a circular annulus of inner radius  $a_1$  and outer radius  $a_2$ ,  $\gamma_a$  is related to  $p_c$  by:

$$\gamma_a = \frac{p_c^2 a_1^2}{32 G_1} \left[ \frac{\delta^2 - 1}{\ln \delta} - 2 \right]^2 \quad (7)$$

where  $\delta$  is defined as the annular ratio  $a_2 / a_1$ . Although approximate, it is instructive to examine the limiting behavior of equation (7). As  $\delta$  approaches unity,  $\gamma_a$  approaches zero (since no film is exposed, no adhesion can be measured even at infinite pressure), while as  $\delta$  approaches infinity,  $\gamma_a$  gets large for any pressure  $p_c$ . Thus, it is theoretically possible to measure large  $\gamma_a$  values at pressures less than the ultimate tensile stress of the film by making the center island sufficiently small.

Concentric square island structures have been fabricated with an outer size ( $2a_2$ ) of 10 mm and inner size ( $2a_1$ ) of 1 and 2 mm. Smaller  $a_1$  values can be obtained by undercutting the film on a 1 mm island until only 0.25 mm or even 0.125 mm sections of the film remain adhered to the center island (Figure 3b). Peel has been achieved using these underetched structures. Although finite-element analysis of the square island structure will be required to generate accurate values of  $\gamma_a$  from observed debond pressures, order of magnitude values of  $\gamma_a$  can be obtained from equation (7). Preliminary experiments indicate that such values are in the range of 1000-3000 J/m<sup>2</sup>, in fair agreement with values obtained from application of the peel test to thicker films [13].



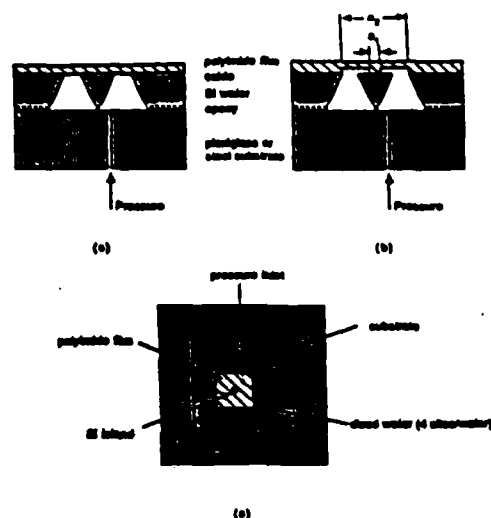


Figure 3. Island adhesion structures.  
(a) side view; (b) side view - undereached film; (c) top view

#### Conclusions

Microfabricated test structures for the *in-situ* measurement of adhesion of thin films have been described. Young's modulus and residual tensile stress are determined from pre-peel measurement of the load-deflection behavior of suspended membranes. On systems of weak adhesion, a blister test using suspended membranes has been carried out. For systems of good adhesion, an island test structure has been developed allowing even thin films to be tested. Mechanical models for all three structures are described.

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